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TRANSMITTAL SHEET

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DATE: February 6, 2006

TO: Ms. Peggy Churchill
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JOB: Libby MT - Guidance Document
Task Order No. 6
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We are sending

herewith..... ☒
under separate cover..... ☐
by messenger..... ☐

1 print(s) each of the following:

Libby MT Asbestos Project: Asbestos Guidance Documents (December 2005)
Volume 1 addendum (Section 9)

Which are

approved..... ☐
approved as noted..... ☐
returned to you for correction and resubmittal..... ☐
for your information..... ☐
for project use..... ☒

The enclosed Section 9 is being replaced in its entirety with the enclosed insert. An updated Table of Contents is also enclosed. Should you have any questions, please call me at 617/452-6309.

Thank you.

Anni H. Autio
Anni H. Autio, P.E.

2006 FEB 14 PM 2:13
EPA REGION 8
SUPERFUND BRANCH

Volume 1: Contents

Section 1

ASTM. 1995. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D 5755-95. American Society for Testing and Materials. October 1995.

Section 2

ASTM. 2001. Standard Practice for Asbestos Detection Limit Based on Counts. ASTM D 6620-00. American Society for Testing and Materials. March 2001.

Section 3

International Organization for Standardization. 1995. Ambient Air – Determination of Asbestos Fibres – Direct Transfer Transmission Electron Microscopy Method. ISO 10312:1995(E).

Section 4

International Organization for Standardization. 1999. Ambient Air – Determination of Asbestos Fibres – Indirect Transfer Transmission Electron Microscopy Method. ISO 13794:1999(E).

Section 5

NIOSH. 1994. Asbestos and Other Fibers by PCM. NIOSH 7400 (Issue 2). August 15, 1994.

Section 6

NIOSH. 1994. Asbestos (bulk) by PLM. NIOSH 9002 (Issue 2). August 15, 1994.

Section 7

Schneider, Thomas, et al. "Development of a Method for the Determination of Low Contents of Asbestos Fibres in Bulk Material." (European Method). The Analyst. June 1998: 1393-1400.

Section 8

Su, Shu-Chun. Reference Charts for Visual Estimate of Area Percentage in SEM Analysis (For Monitor Screen with Aspect Ratio 1.64). S&L Consulting International: 2002.

Section 9

Syracuse Research Corporation (SRC). 2003. Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy. SRC-LIBBY-03 (Rev. 1). December 11, 2003.

Section 10

Syracuse Research Corporation (SRC). 2003. Measurement of Dust Loading SRC-LIBBY-04 (Rev. 0). June 27, 2003.

Section 11

Syracuse Research Corporation (SRC). 2003. Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy. SRC-LIBBY-01 (Rev. 1). May 20, 2003.

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Syracuse Research Corporation (SRC). 2003. Quantification of Asbestos in Soil by SEM/EDS. SRC-LIBBY-02 (Rev. 2). October 28, 2005.

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Section 15

USEPA. 1987. Interim Transmission Electron Microscopy Analytical Methods — Mandatory and Nonmandatory — and Mandatory Section to Determine Completion of Response Actions (AHERA). Final Register dated March 26, 2002.

Section 16

USEPA. 1990. Superfund Method for the Determination of Asbestos in Ambient Air (Interim Version) — Part 1: Method. EPA/540/2-90/005a. February 1990.

Section 17

USEPA. 1990. Superfund Method for the Determination of Asbestos in Ambient Air (Interim Version) — Part 2: Technical Background Document. EPA/540/2-90/005b. February 1990.

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USEPA. 1993. Method for the Determination of Asbestos in Bulk Building Materials. EPA/600/R-93/116. July 1993.

Section 19

USEPA. 1994. Determination of Asbestos Structures over 10 μ in Length in Drinking Water. Method 100.2. June 1994.

Section 20

USEPA. 2000. Asbestos Analysis of Soil by Scanning Microscopy and Energy Dispersive X-Ray Spectroscopy. EPA-LIBBY-01 (Rev. 2). August 28, 2002.

Section 21

USEPA. 2000. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials (Interim Version). EPA/540/R-97/028. November 2, 2000.

Section 22

USEPA. 2004. Analysis of Asbestos in Soil by TEM. EPA-LIBBY-03 (Rev. 2). August 10, 2004.

Section 23

USEPA. 2004. Analysis of Asbestos in Soil by TEM Following Water Sedimentation Fractionation. EPA-LIBBY-07 (Rev. 4). August 10, 2004.

Section 24

USEPA. 2003. Sampling and Analysis Plan for Indoor Dust (For Use at the Libby, Montana, Superfund Site). EPA R8 (Rev. 0). August 2003.

Section 25

USGS. 2002. Guide to Analysis of Soil Samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. October 17, 2002.

Section 26

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Insert for Section 9

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE IN LIBBY PE STUDY ONLY

ANALYSIS OF SOIL-LIKE MEDIA FOR ASBESTOS BY POLARIZED LIGHT MICROSCOPY

Date: April 20, 2004

SOP No. SRC-LIBBY-03 (Revision 1)

Title: **ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY**

Author: William Brattin

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy (PLM) is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project-specific modifications intended specifically for use at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8	<u>[Signature]</u>	<u>4/20/04</u>
Syracuse Research Corp.	<u>William J. Brattin</u>	<u>04/20/04</u>

Revision	Date	Principal Changes
0	03/03/03	--
1	12/11/03	Clarify binning assignment of samples at 0.2%

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1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using polarized light microscopy (PLM). This SOP is specifically intended for application at the Libby Superfund site.

2.0 SCOPE AND APPLICATION

This method is intended mainly for analysis of asbestos in soil or other similar soil-like media. This method is appropriate for the analysis of all types of asbestos fibers, including both chrysotile and amphiboles, including those that are characteristic of the Libby site.

3.0 RESPONSIBILITIES

It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance procedures are performed in accord with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis. The laboratory supervisor should also communicate with project managers at EPA or their oversight contractors any situations where a change from the SOP may be useful, and must receive approval from EPA for any deviation or modification from the SOP before proceeding with sample preparation and analysis.

4.0 METHOD DESCRIPTION

The soil sample to be evaluated for asbestos content by PLM is examined under stereomicroscopy and under PLM (3-5 slides per sample) to estimate the amount of asbestos present. Quantification of the amount of asbestos present may be done either using a visual estimation approach or by a point counting approach, as specified in the Chain of Custody request. In either case, the concentration of Libby amphibole asbestos in the sample is estimated in terms of mass fraction (i.e., percent asbestos by weight) based on the use of project-specific reference materials (calibration standards).

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5.0 DETAILED METHOD

5.1 Basic Methods

All qualitative and quantitative analyses are to be performed in general accordance with the methods and techniques specified in NIOSH 9002, EPA 600/R-93/116, and CARB Method 435. Project-specific modification, clarifications, and requirements are provided below.

5.2 Visual Estimation Approach

5.2.1 Classification of Asbestos Mineral Type

Based on fiber attributes (morphology, refractive index, color, birefringence, etc.), asbestos in the sample is classified into one of three categories:

Code	Description	Notes
LA	Libby Amphibole	Refractive index values for LA span the standard values for tremolite/actinolite (EPA 1993), but may include values for other similar amphiboles (e.g., winchite, richterite) characteristic of the mine at Libby. Based on analysis of 4 different samples from the mine (Wylie and Verkouteren 2000; USGS, unpublished data; Verkouteren, personal communication), observed refractive indices of Libby amphiboles range from about 1.629-1.640 γ and 1.614-1.623 α , with a birefringence of about 0.017. The full range of refractive indices of samples from the mine may be somewhat greater.
OA	Other amphibole	Includes amphibole forms (e.g., amosite, crocidolite, anthophyllite) that are not thought to occur in significant amount at the mine in Libby
C	Chrysotile	

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5.2.2 Estimation of LA Mass Percent

The visual area estimation is a semi-quantitative approach that requires the microscopist to estimate the area fraction of the total material present in a field of view that consists of asbestos material. Because this estimation may be difficult, especially at low concentration values, and because the desired output is an estimate of mass fraction (rather than area fraction), all visual estimates of Libby amphibole content will be performed using a set of site-specific reference materials (calibration standards) as a frame of reference. These reference material will contain either 0.2 % or 1.0% Libby amphibole by weight¹, and have been prepared for analysis using the same approach as for field samples. Using the two reference concentrations (0.2% and 1.0%) as a visual guide, the microscopist will evaluate the field sample and report the results as follows:

PLM Laboratory Report			Description
Qual	Conc (wt.%)	Bin	
ND		A	Asbestos was not observed in the field sample
Tr		B1	Asbestos was observed in the field sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	Asbestos was observed in the field sample at a level that appeared to be approximately equal to or greater than the 0.2% reference material but was less than the 1% reference material.
	1, 2, 3, etc	C	Asbestos was observed in the field sample at a level that appeared to equal or exceed the 1% standard. In this case, the mass percent is estimated quantitatively.

"ND" (not detected) in the Qualifier column is used for all samples in which asbestos is not observed under stereomicroscopy and is also not detected in five (5) different PLM slides

¹ The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

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prepared using representative sub-samples of the test material. These samples are assigned to **Bin A**.

"Tr" (trace) in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in at least one out of 3-5 PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be less than the 0.2 % reference material. These samples are assigned to **Bin B1**.

"<" (less than) in the Qualifier column and 1 in the Concentration column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be equal to or greater than the 0.2 % reference material but less than the 1% reference material. These samples are assigned to **Bin B2**.

A numeric value (1, 2, 3, etc) in the Concentration column without an entry in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be similar to or greater than the 1 % reference material. These samples are assigned to **Bin C**.

Note that because these reference materials are based on Libby amphibole, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, other types of asbestos). Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

5.3 Point Counting Approach

5.3.1 Counting Procedure

Any analysis in which evaluation by point counting is requested will be performed in general accordance with the descriptions provided in EPA/600/R-93/116 and CARB Method 435. The total number of particles to be counted (generally 400 or 1000) will be specified in the Chain of Custody request.

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Take eight sub-samples of the soil sample and mount each separately with the appropriate refractive index liquid. The preparations should not be heavily loaded. Each sample should be uniformly dispersed to avoid overlapping particles and allow 25-50% empty area within the fields of view.

An ocular reticule (point array) or cross-hair is used to visually superimpose points on the microscope field of view. Count 1/8 of the total points required on each of the 8 slides (e.g., 50 non-empty points per slide for a 400 point count and 125 non-empty points per slide for a 1000 point count). For each non-empty point counted, assign the particle that is present at the point into one of four bins:

- Not asbestos
- Libby asbestos (LA)
- Other asbestos (OA)
- Chrysotile asbestos (C)

In order for a particle to be counted as asbestos, the aspect ratio must be $\geq 3:1$.

After the required total number of non-empty points have been counted, record the total number of points in the LA, OA and C bins on the point counting data sheet.

5.3.2 *Estimation of Mass Percent*

Like visual estimation, the output of the point counting approach is an estimate of area fraction, not mass fraction. For this site, point-count estimates of area fraction for Libby amphibole particles will be converted into estimates of mass fraction using a standard curve approach.

The standard curve will be prepared using a series of site-specific reference materials (calibration standards) containing 0%, 0.2%, 0.5%, 1%, or 2% Libby amphibole. The area fraction of each reference material will be estimated by the point counting approach in quadruplicate. The standard curve will be prepared by plotting the mean area fraction determined by point counting versus the mass percent in the reference material. The mass fraction of a field sample will be determined by measuring the area fraction of the field sample and locating the mass fraction that corresponds to that area fraction on the standard curve.

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Because the standard curve is based on Libby amphibole, it is not appropriate to utilize this standard curve for other types of asbestos. Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

6.0 APPARATUS AND MATERIALS

Polarized light microscope, with lens and filters
Stereomicroscope (approximately 10-45x)
Petri dish for stereomicroscopic sample examination
Spatula and forceps
Glass slides and cover slips
Refractive Index (RI) oils
Reference Materials (Calibration Standards)
 Soil containing 0.2% LA by mass
 Soil containing 0.5% LA by mass
 Soil containing 1.0% LA by mass
 Soil containing 2.0% LA by mass
Laboratory log book
Data recording sheet (Attachment 1)

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Precision and Accuracy

PLM by visual estimation and point counting are both semi-quantitative methods. For the purposes of this project, the accuracy and precision of the method are evaluated by measuring the frequency with which samples are assigned to the correct "bins". Data on precision and accuracy of bin assignment will be collected in the future and used to establish performance criteria for this project.

7.2 Method Proficiency

At present, sufficient data are not available to establish a quantitative procedure for method proficiency demonstration. As results become available, a procedure will be established and

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applied, based on the analysis of a set of blind Performance Evaluation materials and assessing the frequency of correct bin assignments. If the assignments reported by a laboratory are within acceptance criteria bounds (see Section 7.1), then that laboratory will be deemed proficient. If not, remedial actions must be taken to address the errors before work may begin by that laboratory.

8.0 RECORDS

8.1 PLM Data Forms

Analysts will record analytical results using the electronic data sheets developed for the Libby project, as presented in Attachment 1. Note that there are two different electronic forms; one is for use in visual area estimation, and the other is for use in point counting. Once completed and checked, these spreadsheets are submitted to EPA for upload into the database. The laboratory should retain all original records for use in resolving any questions until otherwise instructed by EPA.

8.2 Instrument Maintenance Logbook

An individual instrument maintenance logbook should be kept for each piece of equipment in use at the laboratory. All maintenance activities must be recorded in the appropriate logbook.

8.3 Data Storage and Archival

Electronic Data. Each day of data acquisition, all electronic files will be saved onto two separate media. For example, the data may be saved onto a computer hard drive, but must also be backed up onto a type of portable media such as CD-ROM, floppy disc, or tape. Portable media will be maintained in a single location with limited access.

Hardcopy Data. All data sheets and micrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

Copies (hardcopy and electronic) of the raw analytical data will be submitted to USEPA for archival.

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9.0 REFERENCES

CARB 435. California Environmental Protection Agency Air Resources Board, Method 435, Determination of Asbestos Content in Serpentine Aggregate. June 6, 1991.

EPA. 1993. Method for the Determination of Asbestos in Bulk Building Materials. United States Environmental Protection Agency, Office of Research and Development. EPA/600/R-93/116. July 1993.

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ATTACHMENT 1

PLM DATA RECORDING SHEETS

PLM (VE and PC) Data Sheet and ED.xls

(Check with Volpe or SRC to determine the latest version number)

PLM VISUAL ESTIMATION DATA RECORDING SHEETPage of

Laboratory Name

Job Number

Date Received _____

SOP Name/Revision

[illegible]

Comments (Use back if needed)

PLM POINT COUNT DATA RECORDING SHEET

Page ____ of ____

Laboratory Name

Job Number

Date Received _____

SOP Name/Revision

[illegible]

Comments (use back if needed)